Analysis of Rock and Sediment Standard Prepared in Japan by Neutron Activation and X-ray Fluorescence Methods (Commemoration Issue Dedicated to Professor Tsunenobu Shigematsu on the Occasion of his Retirement)

AUTHOR(S):

Takamatsu, Takejiro; Kawashima, Munetsugu; Koyama, Mutsuo

Bull. Inst. Chem. Res., Kyoto Univ., Vol. 58, No. 2, 1980

Note

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Takejiro TAKAMATSU, Munetsugu KAWASHIMA, and Mutsuo KOYAMA*

Received March 1, 1980

Instrumental neutron activation analysis and X-ray fluorescence anaysis were performed on rock and sediment standard or reference materials (SRM) prepared in Japan. The rock SRM in called JG-1 prepared by Geological Survey of Japan and the sediment SRM is Sanshiro Pond Sediment prepared by National Institute for Environmental Research.

KEY WORDS : SRM / Neutron activation analysis / X-ray fluorescence analysis / Neutron spectrum sensitive monitors /

1) Neutron Activation Analysis.

200 mg of samples in average were weighed in sample containers made of clean polyethylene for one hour irradiation. 20 to 30 mg of samples were taken in a similar manner for five minutes irradiation. Before packing weighed samples into an irradiation capsule, those containers were wrapped with clean polyethylene bags in order to reduce chances to be contaminated during handling and irradiation. The standard for one hour irradiation was made by spotting standard solution containing ten to fifty micro grams of Co, Cr, Sb and U on the same sheet of Millipore Filter and air dried. With the combination of these elements, neutron spectrum can be evaluated. After packed in sample containers, standards were placed at the central part of irradaition capsules so as to minimize the error caused by the neutron flux gradient within the irradiation capsules. The error caused by the flux gradient would be estimated to be ± 5 percent at most by this arrangement. The standard for five minutes irradiation was made by spotting Na and Mn solutions.

Neutron irradiation was performed with a pneumatic facility average flux and spectrum of which are described elsewhere.[1] Samples irradiated for five minutes were cooled for two to three hours and those irradiated for one hour cooled for eight to ten days. γ-ray spectra were measured with a Ge(Li) detector of the active volume of 53 cc coupled to a NAIG-D 4K channels MCA which was equipped with an open reel magnetic tape out put. All the data recorded on magnetic tapes were treated by a minicomputor OKITAC 50. Calculations were made by using nuclear data such as

* 高松武次郎, 川島宗継, 小山睦夫: Research Reactor Institute, Kyoto University, Sennan-gun, Osaka, Japan.

Table I. Neutron Activation and X-ray Fluorescence Analysis of JG-1 and Sanshiro Pond Sediment

Element	Nuclide	γ-ray (kev)	JG-1 Determined Content	JG-1 Agerage	JG-1 Reported Range	Sanshiro Pond Sediment Content	Method
Fe	Fe -59	1099.0	1.45 ± 0.05 (9) %	15.3 %	1.36 ~ 1.76%	6.37 ± 0.12 (10) %	NAA
Na	Na -24	1368.6	2.46 ± 0.11 (5)	2.51	2.33 ~ 2.66	0.537 ± 0.014 (10)	NAA
K						0.715 ± 0.018 (15)	XRF
Ca						0.620 ± 0.011 (14)	XRF
Ti						0.673 ± 0.013 (15)	XRF
Sm	Sm -153	103.18	5.60 ± 0.4 (7) ppm	4.6 ppm	4.15 ~ 5.6 ppm	5.17 ± 0.14 (9) ppm	NAA
Ce	Ce -141	145.43	55 ± 6 (9)	43.2	41.9 ~ 47	41.2 ± 1.6 (10)	NAA
Lu	Lu -177	208.34	0.64 ± 0.08 (7)	0.36	0.22 ~ 0.52	0.30 ± 0.02 (10)	NAA
U	Np -239	277.63	3.53 ± 0.18 (5)	3.3	2 ~ 4.67	1.66 ± 0.25 (10)	NAA
Th	Pa -233	311.98	14.5 ± 1.20 (5)	13.5	11.5 ~ 15.6	5.77 ± 0.27 (10)	NAA
Cr	Cr -51	320.07	69.7 ±20.0 (5)	52.7	31 ~ 64	74.2 ± 3.8 (10)	NAA
Hf	Hf -181	482.0	3.59 ± 0.25 (9)	3.3	3.1 ~ 3.8	3.55 ± 0.31 (10)	NAA
Yb	Yb -169	197.95	3.08 ± 0.40 (7)		1.45 ~ 2.5	2.14 ± 0.19 (10)	NAA
Au	Au -198	411.8	— —	—	—	0.09 ± 0.01 (10)	NAA
Ba	Ba -131	496.23	508 ±60 (5)	462	430 ~603	324 ±56 (10)	NAA
Sb	Sb -122	564.10	0.66 ± 0.20 (3)		0.1 ~ 2.0	2.35 ± 0.12 (10)	NAA
As	As -76	559.1	— —	—	— —	10.5 ± 1.0 (10)	NAA
Br	Br -82	776.5	— —	—	— —	15.3 ± 0.7 (5)	NAA
Cs	Cs -134	795.76	10.3 ± 0.50 (5)	10.1	9.3 ~ 10.6	3.87 ± 0.42 (10)	NAA
Mn	Mn-56	846.6	489 ±13 (3)	472	433 ~557	787 ± 8 (5)	NAA
Sc	Sc -46	889.25	6.36 ± 0.26 (9)		6.44 ~ 8.0	26.3 ± 0.4 (10)	NAA
Rb	Rb -86	1078.80	192 ±15 (5)	181.3	171 ~202	47.6 ± 5.9 (10)	NAA
Co	Co -60	1173.21	3.05 ± 0.30 (9)	6.4	2 ~ 28	26.6 ± 0.9 (10)	NAA
Ta	Ta -182	1221.38	— —	—	— —	[0.4]	NAA
Eu	Eu -152	1408.02	0.46 ± 0.03 (7)	0.69	0.62 ~ 0.75	0.89 ± 0.06 (10)	NAA
La	La -140	1596.40	23.2 ± 1.9 (7)	22.1	18 ~ 22.5	18.0 ± 0.6 (10)	NAA
Ni						31 ± 3 (10)	XRF
Cu						236 ± 2 (10)	XRF
Zn						379 ± 6 (10)	XRF
Sr						113 ± 2 (10)	XRF
Pb						118 ± 1 (10)	XRF

NAA: Neutron activation analysis, XRF: X-ray fluorescence analysis
( ): Number of determinations, [ ]: Rough estimation
The contents of the Sanshiro Pond sediment are based on the material dried at 110°C for 4 hrs.

cross sections, resonance integrals, halflives, etc. stored in a file, after evaluating neutron spectrum with monitors irradiated together with samples.[1]

2) X-ray Fluorescence Analysis.

X-ray fluorescence analysis was performed by a non-destructive method which utilizes internal standards such as Se and Cs of known amounts preliminarily well mixed with samples. Details are already reported elsewhere.[2]

Both the methods described above do not require individual primary standards corresponding to elements to be determined in samples. Therefore, chance errors on making complicated mixtures can be eliminated.

3) Result.

In Table I results are shown. In case of JG-1, GSJ have been compiling data obtained by different analytical methods and from people from different disciplines.[3] For reference, recommended values by GSJ and ranges of reported values are cited. Pretty good agreements were obtained for most of the elements except for the cobalt concentration. Since cobalt is one of elements which are easily and accurately determined by neutron activation analysis, the value presented in this work is more accurate than the recommened. Chromium concentrations determined by neutron activation sometimes become twice as much as that of the recommended. The reason for this is not clear. Contamination of the elements during the sample preparation and irradiation is very unlikely, since blank runs so far examined never exhibited significant amounts of those elements listed in Tables.

Concentrations listed for Sanshiro Pond Sediment are corrected for 10.4 percent of the water content which was measured independently by drying samples at 110°C. Good reproducibility of the analytical data of the SRM suggests that the sample is homogeneous enough to be used to the practical purpose.

REFERENCES

(1) M. Koyama and R. Matsushita, *Bull. Inst. Chem. Res., Kyoto Univ.*, **58**, 235 (1980).
(2) T. Takamatsu, *Bunseki Kagaku*, **27**, 193 (1978).
(3) A. Ando, H. Kurasawa, T. Ohmori, and E. Takeda, *Geochem. J.*, 8, 175 (1974).